Correlative Compositional Analysis of Fiber-Optic Nanoparticles

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H. Francois-Saint-Cyr[1], I. Martin[1], W. Blanc[2], P. LeCoustumer[3], C. Hombourger[1], D. Neuville[4], D. J. Larson[1], T. J. Prosa[1], C. Guillermier[5]

[1] CAMECA Instruments Inc., 5500 Nobel Drive, Suite 100, Madison, WI, 53711, USA
[2] Université Nice Sophia Antipolis, CNRS, LPMC, UMR7336, 06100 Nice, France
[3] Université Bordeaux 3, Géo-ressources et Environnement, EA4592, 33607 Pessac, France
[4] Institut de Physique du Globe de Paris, 1 rue Jussieu, 75005 Paris, France
[5] National Resource for Imaging Mass Spectroscopy, Cambridge, MA 02139, USA

Development of new active optical fiber devices requires materials with augmented intrinsic properties, using luminescent ion–doped silica as a host glass. Nanoparticles (NPs) in optical fibers can provide this augmentation as they can combine the sturdiness and low cost of silica with particular spectroscopic behavior that would not appear in a pure silica local environment. Ideally, NPs would fully encapsulate luminescent ions to produce engineered spectroscopic properties. This technology would be of great interest for a large domain of applications: high power fiber lasers, random lasers, light sources with new wavelengths and telecommunications.

As silicate systems have a large phase immiscibility domain when they contain divalent metal oxides (such as Mg), one can take advantage of thermal treatments inherent to the MCVD process to obtain NPs through phase separation. NPs are grown *in situ* within the material. Although glass-ceramics were discovered in 1950's, there is a great demand on experimental data to understand the early stages of nucleation [1]. Modern glass ceramics are generally obtained through many trial-and-error processing steps, involving variations in composition as well as thermal treatments. Although classical nucleation theory was the first model proposed to explain those phenomena, growth rate mismatches could encompass several order of magnitude. According to this capillary assumption-based model, nuclei properties are the same as those of the bulk (same structure and composition). Recent articles disprove assumption of structure, pointing toward NPs structural changes [2] and transition from amorphous nuclei to crystalline NPs [3]. Compositional changes for small particle sizes (~1-10 nm) have been measured in alloys with Anomalous Small Angle X-Ray Scattering (ASAXS) [4] and in steels with Atom Probe Tomography (APT) [5]. Recent developments in APT allowed the extension of such studies to glass-ceramics, presenting additional challenges in specimen preparation and yield [6]. In the current work, we report experimental data disproving the second capillary assumption at the early stage of nucleation-growth process.

Composition of NPs was investigated using APT, NanoSIMS and Transmission Electron Microscopy. The Mg distribution in silica-based glass doped with Mg, P, Ge and Er is reported in Figure 1. When NPs sizes range in the 1-10 nm domain, we can clearly quantify the NPs concentration of Mg, P and Er (Figure 2). These nucleation and growth mechanisms in optical fibers impact the overall material community as experimental results could refine the theories behind these processes.

References

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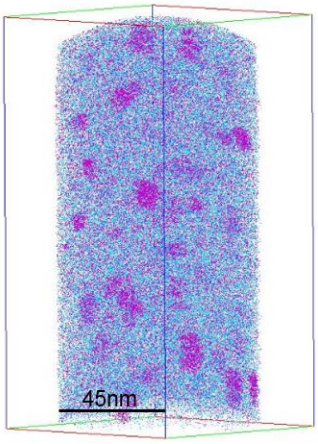

Figure 1. Mg-based dielectric nano-particles (pink) surrounded by silica matrix (blue).

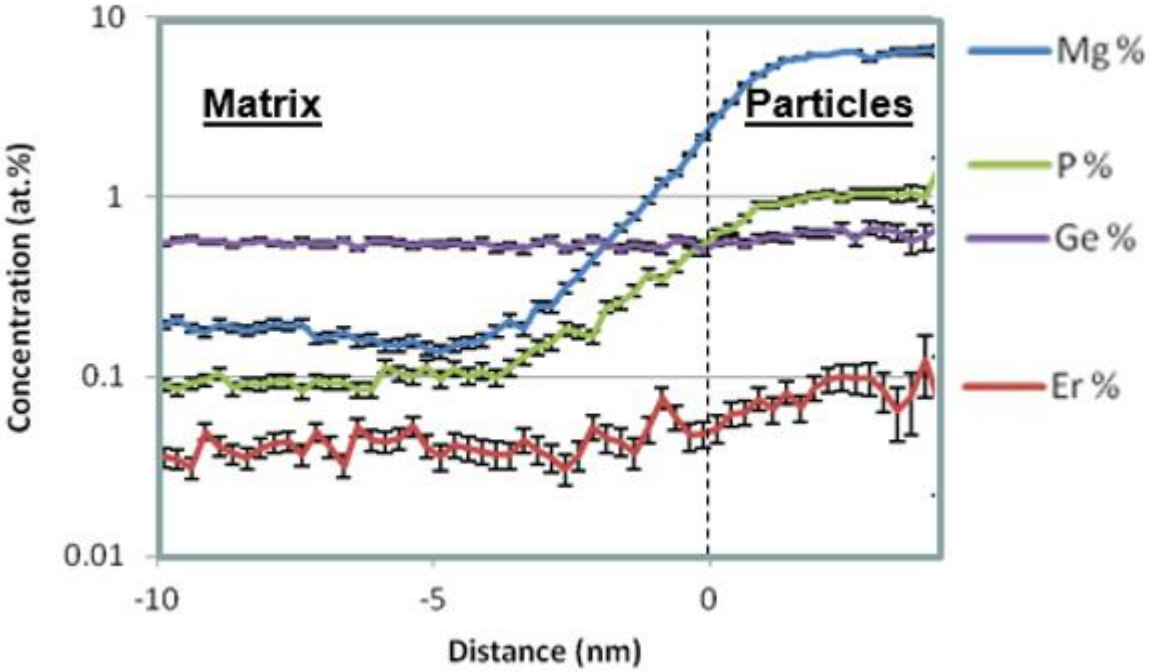

Figure 2. Proximity histogram displaying the evolution of Mg, Er, P, and Ge concentrations from the silica matrix toward the center of the Mg-based dielectric nanoparticles.